Novel Micro-Plastics Extraction Line: Elutriation, Chemical Digestion, and Density Separation

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Introduction

The separation of microplastics from ocean water samples is a highly researched topic in the marine science field. Several papers have investigated methods to successfully collect and quantify microplastic contamination in our ocean basins. Little to no papers have researched the collection of microplastics from marsh substrate/sediment samples due to accessibility and cohesiveness. The Winyah Bay area is under high environmental stress from high influx of the local fisheries tourism and also the plastic manufacturing industry that is located around the Georgetown area. We have found plenty of industrial pellets collected in and around these waterways. This not only has adverse effects on humans but also has detrimental marine life.

Area of Study

This study is focused in the coastal marsh regions of South Carolina. Specifically the Winyah Bay region located in Georgetown County, SC (Fig. 1). The Waccamaw, Great Pee River and the Sampit River all feed into Winyah Bay. We have located several assumed point sources contributing to microplastic contamination have been within this region causing marine debris to enter Winyah Bay as well as the Atlantic Ocean.

Figure 1: A map of Winyah Bay, the boxes represent the three different river that feeds into Winyah bay. The Waccamaw in white, The Great Pee Dee in yellow and The Sampit in green.

Hypotheses

Through the optimization of the three step microplastic extraction line, microplastic contamination in the coastal area will be prepared for the following identification and quantification step with the aim to reduce through the location of sources.

Occupy the knowledge gap between highly researched methods of microplastic extraction and separation from oceanic water samples with the unknown techniques of marsh samples to determine:

- **Optimize a elutriation column that will successfully suspend and separate microplastics attached to marsh sediments.**
- **Consider the best chemical process and reaction parameters to completely degrade organic matter within the sample without corroding any plastic particles.**
- **Final, highly enriched microplastic sub-samples, also presorted regarding their own density.**

Objective

The development and optimization of a 3-step microplastic extraction line using elutriation, chemical digestion and density separation toto disintegrate, separate, and concentrate the microplastic content smaller than 5 mm within highly cohesive, organic-rich, heterogeneous sediment samples from tidal and marsh systems. The ultimate goal is a fully qualitative and quantitative of microplastic loading.

Challenges

- Marsh substrate/samples are extremely cohesive and contain lots of organic material which microplastics like to bind to.
- Building an efficient, affordable, and effective technique is crucial for evaluating microplastic contamination within our region.
- Must successfully extract all microplastic particles from marsh samples by suspending plastic material, effectively separating and digesting all organic material without the over digestion of any plastics.

Methods

- Sufficient sediment sample collection in the field.
- Development and optimization of three-step microplastic extraction line.
- Evaluate microplastic contamination and quantify microplastic loading in wetland areas of Georgetown, SC.

Extraction Process

Step one: Material Elutriation

The first step in this microplastic extraction process is most crucial to the suspension of particles and separation of cohesive marsh sediments; Elutriation. The column we constructed was modeled after a study done by Claessens et al. (2013). (Fig. 2). Several key features give the column its unique ability to stand apart from previously constructed models.

- **Assembled by mostly PVC pieces allows for the column to be efficiently cleaned following elutriation and ensure no water leakage.**
- **The column holds 47.0 L of water, filled by a water pipe attached to the bottom.**
- **Air-stone located within the column that is connected to an air compressor, used to generate an airflow for effectively suspending the sediment.**
- **Overflow valve at the top to capture the very surface layer of the water collecting, trapping and filtering microplastics .**

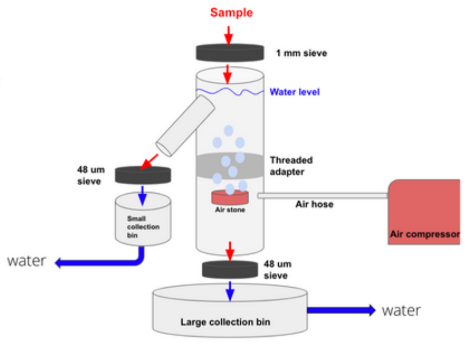

Figure 2: A rough schematic of the elutriation column created by our team.

Standardized Procedure:

1. **Water samples are pre-treated with Sodium Hexametaphosphate ($Na(PO_3)_6$) ; common name "Calgon", to lower the electrostatic surface charge and aid in more sediment suspension.**
2. ***300 mL* (wet volume) of marsh sample was added to column**
3. **Column filled with tap water 3/4 fill-line**
4. **Air compressor turned on for 5 minutes**
5. **Settling Period - 5 mins**
6. **Overflow Period - 5 mins.**
7. **Each sample repeatedly treated 3 times.**

Step two: Chemical Digestion

Continuing along the multi-step microplastic extraction line, the overflow collected post elutriation is passed onto a chemical digestion process. The sample is run through an optimized chemical treatment for a fully controlled, mild, yet effective digestion of any residual bio-organic plant matter left in the sample.

Fenton's Reagent is an effective chemical treatment utilized to aid in the formation of hydroxyl radicals which are the key component in the breakdown of organic matter. High reactivity of these hydroxyl radicals is achieved through the combination of Ferrous (Fe^{2+}) or Ferric (Fe^{3+}) Ions and Hydrogen Peroxide (H_2O_2). Since even milder acids may lead to corrosion of some plastics types, we maintain full control over the reaction temperature and acidity during the whole digestion process.

The optimization of certain parameters such as temperature, pH, H_2O_2 concentration, and reaction times are key when applying Fenton's Reagent to these samples. Considering and tampering with these parameters allowed for a desirable, effective but cautious digestion process of OGM and a highly concentrated, organic-free microplastic compound ready for the following density separation.

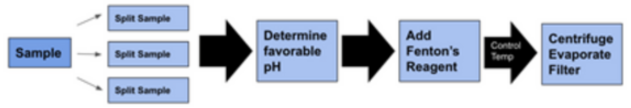

Figure 2: A flow chart for the process of chemical digestion. Each sample goes through the same prccess after being split.

Desirable Parameters Optimization

- **pH of 3.5-4 adjusted by addition of 0.10 M Sulfuric Acid H_2SO_4**
- **Temperature control 4 - 50 oC**
- **Amount of catalyst solution ($FeSO_4 * 7H_2O$) to meddle with reaction rate**

Considerable Factors

- **Contamination of lab space (air particulates, lab attire, no cross-contamination of plastic particles)**
- **All reactions controlled in fume hood and covered against secondary contamination**
- **Constant temperature control checks with thermometers and ice bath (endothermic oxidation reaction)**

Step three: Density Separation

The final step of this novel microplastic extraction process is to separate plastics based on their densities. Most microplastics are lighter than freshwater but some types are denser, it is extremely difficult to quantify all fragments based off of samples treated only to elutriation and chemical digestion. This separation technique utilizes fluids of various density to help separate plastic particles (Fig. 4).

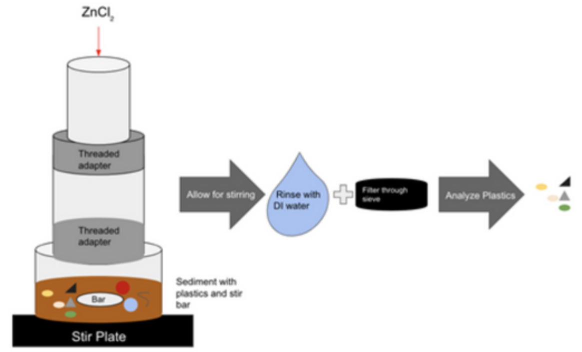

Figure 4: A schematic of the density column. and the process associated with sorting microplastics based on density.

- Following chemical digestion, the sample is placed in the base of a density separation column (Fig. 4).
- Several columns will successively used with varying densities of salt water and some with Zinc Chloride (ZnCl).
- A stir bar is located at the bottom of the column to help stir up the sediment to keep it in suspension for a full exposure to the fluid and respond due to their density.
- The column is then left to rest to ensure the plastics will travel to the fluid surface to be captured.
- Any plastics still on the bottom will be given a higher density solution until all plastics are extracted.

Conclusions

- Successfully built 6 ft. PVC elutriation column capable of suspending cohesive materials within fleshy Marsh water and sediment samples.
- Standardized column procedure to five minute intervals with a constant flow of air pressure (10 psi).
- Run each sample three times, each successive run yielded less fine-grained material were filtered and trapped in the outflow sieve at the bottom of the column. Plastics were located in the top sieve which were capture following the five minutes of overflow.
- Through trial and error of elutriation procedure a greater settling phase of the sample was deemed necessary to allow all sediments to completely settle or lift occur to their specific density before starting the over flow process.
- Fenton's Reagent was considered the most efficient chemical digestion procedure to utilize the highest yield of microplastic particles and affectively digesting organic matter.
- It was discovered in the density separation that it is required to use multiple density fluids. This is due to the wide range of densities of plastics Salt water an extremely versatile density fluid and was chosen to be used for plastics less dense than 1.0-1.2 (g/cm3). While the Zinc Chloride is used for all plastics >1.2 g/cm3 and all plastics </= 1.5-1.7g/cm3.

Citations:

Claessens, M., Cauwenberghe, V., Vandegehuchte, L., Janssen, M.B., C.R., 2013. New techniques for the detection of microplastics in sediments and field collected organisms. Mar. Pollut. Bull. 70(1-2): 227-233.

Oswald, Matthew Comprehensive Manual for the Construction and Operation of an Elutriation Column. 399 Undergraduate research project. Elutriation Manual Version 1 2020.

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